Standard Operating Procedure for Sample Preparation and Analysis of PM10 and PM2.5 Samples by Scanning Electron Microscopy

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TABLE OF CONTENTS

Section **Page**

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1.0 Procedural Section

1.1 Scope and Applicability

The scanning electron microscopy (SEM) standard operating procedure (SOP) establishes procedures for determining the type and amount of particulate matter deposited on a Teflon filter following collection of a PM2.5 or PM10 sample. The SOP is also applicable to particulate samples collected on other filter media such as polycarbonate filters. Particles are analyzed by SEM and by energy-dispersive x-ray spectroscopy (EDS) to determine size, morphology, and particle chemistry.

1.2 Summary of Method

PM2.5 filters are examined by microscopy by first evaporating a thin layer of carbon onto the surface of the sample. The carbon-coated filters are stored for subsequent examination by SEM and EDS. Particles on the sample filter are found using SEM; upon locating a particle, its size and morphology are recorded. The spectrum of the particle is then obtained using EDS. The EDS spectrum is used to determine the elemental composition of the particle. This procedure is repeated for a minimum of 100 particles per filter.

1.3 Definitions

SEM: A microscope which creates an image of a sample by scanning the sample with an electron beam. Secondary electrons are subsequently emitted from the sample, are collected in the microscope detector, and are reconfigured at various magnifications on a cathode ray tube as an image of the sample.

EDS: An instrument which collects x-rays emitted from a sample which has been bombarded by an electron beam. The x-rays are sorted by energy level, and a spectrum of x-ray energy vs. frequency is plotted which is indicative of the elements present in the sample and of the concentration of each element present.

1.4 Health and Safety Warnings

Operators must use care around the vacuum evaporator to avoid hazards inherent in high vacuum devices (e.g., implosion of glassware under high vacuum). Because the SEM instrument employs high voltages, the operator should avoid touching conductors, and should observe and report any damage to electrical insulation or other damage to the instrument. The SEM must be checked regularly for x-ray leakage.

1.5 Cautions

Items used during sample preparation such as petri dishes, forceps, scalpels, stubs, and glassware are critically cleaned prior to use, and prior to contact with subsequent samples. Reagent aliquots used for any given sample preparation must not be used for subsequent samples. The area in which the filters are prepared must be kept as contamination-free as possible, facilitated by the use of a laminar flow clean bench, the use of a fume hood during prep stages requiring volatile chemicals, and the wet-wiping of all counter tops prior to sample preparation. All prep instruments and tools must be quarantined from other areas of the laboratory, particularly where particulate samples are analyzed or stored.

1.6 Interferences

Interferences include particulate contamination which exists on the Teflon filters prior to sample collection, contamination to the filter subsequent to sample collection, and the inherent particulate appearance of the Teflon filter. Filter lots must be characterized prior to use to determine the type and concentration of particulate on the filter. Analysis of field blanks will assist with determination of the potential level of post-collection contamination.

1.7 Personnel Qualifications

Operation of the SEM/EDS instrument requires a high degree of training and skill. The laboratory supervisor will typically have a Master’s degree in chemistry, mineralogy, surface science, or related area. At a minimum, analysts should have a Bachelor's degree in chemistry or related area, and must also receive extensive classroom and hands-on training. All RTI personnel performing SEM/EDS analyses for the PM2.5 program will receive necessary on-the-job training from the laboratory

supervisor. Graduate-level course work and/or continuing education relevant to the analytical technique is strongly encouraged.

1.8 Apparatus and Materials

- Hitachi S-800 scanning electron microscope
- Oxford-Link energy dispersive x-ray spectrometer
- Aluminum SEM specimen stubs
- Double-sided carbon adhesive specimen mounts
- Forceps, scalpels, petri dishes (90mm), 100-ml. beakers

1.9 Calibration

Energy dispersive x-ray spectrometer (EDS) energy levels are calibrated each day of use by checking copper Lα and aluminum Kα peaks prior to use. Minor variation (±10 eV) requires calibration; variations greater then 10 eV may require major service. The magnification of the SEM must be calibrated quarterly using a magnification calibration stub to determine that magnification settings are accurate at the magnification ranges used for PM analysis.

1.10 Sample Collection

Samples received from a client are checked for complete shipment and inclusion of all identification data. Sample identifications are recorded in the project file and the laboratory notebook. In any situation where data is missing or sample validity is in question, the client is contacted before proceeding.

1.11 Handling and Preservation

No special preservation considerations apply. See the next section for a description of sample handling during preparation and analysis.

1.12 Sample Preparation and Analysis

The sample housing is cleaned with a damp wipe, and a SEM substrate (stub) is prepared for receiving a filter by applying a double-sided sticky conductive carbon pad to the stub surface. The underside of the stub is labeled with the sample number using a permanent marker. The filter is removed and placed on the stub, and the stub is placed in a vacuum evaporator for carbon coating. A thin layer of carbon is evaporated onto the surface of the sample at a vacuum of 5.0×10^{-5} torr. The filter is removed and placed in a clean polycarbonate storage box for transfer.

Examine the sample in the SEM as follows:

1. Turn on display power
2. Mount sample
3. Check chamber geometry
4. Check that the sample exchange chamber (SEC) lever is closed
5. Air the SEC
6. Install the sample
7. Evacuate the SEC
8. Open the SEC lever
9. Place the sample in the column
10. Remove the sample holder
11. Close the SEC lever
12. Open the main valve
13. Turn on the high voltage (HV)
14. Adjust brightness, magnification, and focus
15. Center the objective aperture
16. Stigmate the electron beam (compensate for astigmatism by adjusting the symmetry of the electrical fields that focus the electron beam)
17. Screw in the EDS detector
18. Lower sample to 25 mm
19. Call up the ISIS program
20. Log in
21. Collect and print spectra
22. Turn off the HV
23. Close the main valve

24. Restore sample geometry
25. Remove the sample
26. Close the SEC lever
27. Briefly evacuate the SEC
28. Close the SEC vacuum valve
29. Turn off the display power
30. Crank out the EDS detector
31. Close out the EDS computer

When examining the sample, select randomly located areas for higher magnification scanning. Scan specific area for particulate matter. Upon location of a particle, record its size and morphology, and collect a spectrum with the EDS. Either print the spectrum or record pertinent characteristics of the spectrum on a piece of paper. Repeat this procedure for a minimum of 100 particles encountered, regardless of size, morphology, or chemistry.

1.13 Troubleshooting

Because of the highly technical nature of the SEM and EDS measurements, the reader is directed to the Hitachi S-800 Operations Manual for troubleshooting advice. All troubleshooting should be done by qualified personnel.

1.14 Data Acquisition, Calculations and Data Reduction

Report size distribution of the particles measured, any notable morphological characteristics of the particles, and the chemical characteristics of the particles. The report does not need to include a spectrum for each particle, though it should include spectra representing each general type of particle found. Size distribution is reported using a table of particle sizes and a histogram of particle size versus frequency.

1.15 Computer Hardware and Software

The Oxford-Link EDS will assist with the identification of elemental peaks on the spectrum, but does not automatically assign identities to peaks. There are no software decisions made in the analytical process or automated functions performed by the SEM or EDS. Size distribution graphs

included in the report are generated by inputting data into a spreadsheet software program, which automatically plots a graph of the size frequency distribution.

1.16 Data and Records Management

Each project is kept in a dedicated labeled folder in chronological order in a secure office location. All records are retained for a minimum of seven years.

2.0 Quality Control

Various quality control (QC) checks are performed to ensure analytical quality. These checks are performed on sample preparation equipment, supplies, laboratory areas, and analytical instrumentation. The chief ongoing QC check is related to instrument calibration, described in Section 1.9 above. Field blanks submitted with project batches will be prepared and analyzed as standard samples.

3.0 References

Hitachi S-800 Operation Manual